

PATENT ABSTRACTS OF JAPAN

(11)Publication number : 11-217792

(43)Date of publication of application : 10.08.1999

(51)Int.Cl.

D21H 17/37

(21)Application number : 10-046157

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(22)Date of filing : 21.01.1998

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(54) ADDITIVE FOR PAPERMAKING AND PAPERMAKING

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain an additive for papermaking, capable of manifesting activities for improving a yield and a paper strength within a wide pH region by adding a twitterionic acrylamide-based copolymer consisting essentially of a primary alkenylamine and/or a salts thereof to a pulp slurry as the additive.

SOLUTION: This additive for papermaking of a twitterionic acrylamide-based copolymer is obtained by polymerizing a vinyl monomer mixture comprising 0.01-20 mol.% primary alkenylamine and/or salts thereof, e.g. 2-propene-1-amine, 40-98.99 mol.% acrylamides, 0.5-20 mol.% anionic vinyl monomer such as acrylic acid and 0.5-20 mol.% cationic vinyl monomer such as diethylaminoethyl acrylate. The additive for the papermaking of 0.01-10 wt.%, preferably 0.03-3 wt.% based on the bone-dried solid component of the pulp is added to the pulp slurry and the pulp slurry with the added additive is subjected to the papermaking.

LEGAL STATUS

[Date of request for examination]

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

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CLAIMS

[Claim(s)]

[Claim 1] The additive for paper manufacture characterized by consisting of both ionicity acrylamide system copolymer that uses a first-class alkenyl amine and/or its salts (a) as an indispensable component.

[Claim 2] The additive for paper manufacture according to claim 1 which is both the ionicity acrylamide system copolymer obtained by both the ionicity acrylamide system copolymer carrying out the polymerization of the vinyl monomer mixture which comes to contain a first-class alkenyl amine and/or its salts (a), acrylamides (b), an anionic vinyl monomer (c), and a cationic vinyl monomer (d).

[Claim 3] A first-class alkenyl amine and/or the additive for paper manufacture of the salts (a) according to claim 1 or 2 whose a kind is a 2-propene-1-amine at least.

[Claim 4] both -- ionicity -- acrylamide -- a system -- a copolymer -- the first class -- the alkenyl -- an amine -- and/or -- the -- salts -- (-- a --) -- 0.01 - 20 -- a mol -- % -- acrylamide -- a kind -- (-- b --) -- 40 - 98.99 -- a mol -- % -- anionic -- vinyl -- a monomer -- (-- c --) -- 0.5 - 20 -- a mol -- % -- cationicity -- vinyl -- a monomer -- (-- d --) -- 0.5 - 20 -- a mol -- % -- containing -- becoming -- vinyl -- a monomer -- mixture -- a polymerization -- carrying out -- obtaining -- having had -- both -- ionicity -- acrylamide -- a system -- a copolymer -- it is -- a claim -- one -- two -- three -- either -- a publication -- paper manufacture -- ** -- an additive -- .

[Claim 5] The paper manufacture approach characterized by adding and carrying out paper making of the additive for paper manufacture of a publication to a pulp slurry at either of claims 1, 2, 3, and 4.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the additive for paper manufacture and the paper manufacture approach of consisting of both ionicity acrylamide system copolymer set and used like a paper maker. Furthermore, it is related with the new additive for paper manufacture and the paper manufacture approach of discovering the filterability which was excellent in detail also in the paper-making conditions in neutral thru/or alkaline broad pH field from acidity, the yield, and a paper durability enhancing effect.

[0002]

[Description of the Prior Art] the increment in the used paper utilization factor set like the paper maker of paper and the paper board, and according to factors, such as aggravation of a bolt supply situation, and energy saving, and closing-izing of a paper-making system -- progressing -- quality of paper -- when planning improvement and a productivity drive, the importance of a paper reinforcing agent, a filtration improvement agent, and a yield improver increases, and these various additives for paper manufacture are offered.

[0003] As an additive for paper manufacture, starch, a polyamide polyamine epichlorohydrin resin, polyacrylamide system resin, etc. are used widely, and use of polyacrylamide system resin is expanded especially. As a class of additive for paper manufacture which consists of polyacrylamide system resin, there are an anionic polymer, a cationic polymer, and both ionicity polymer greatly from the difference in the ionicity. Also under the paper-making conditions that both the ionicity copolymer obtained by aggravation of the paper-making raw material of this inside or recent years, improvement in a paper-making rate, etc. by copolymerizing an anionic vinyl monomer and a cationic vinyl monomer with acrylamide (meta) is severe, since there are comparatively few falls of effectiveness, the rate of an use rate has increased. However, these linear types of polymer is also inadequate for corresponding to a demand of high quality which was described above, aggravation of paper-making conditions, etc.

[0004] It faces carrying out the polymerization of acrylamide (meta), a cationic vinyl monomer, and the anionic vinyl monomer, and preparing an acrylamide system copolymer recently, and the acrylamide system copolymer which has the high branching structure of the amount of macromolecules which introduced the cross-linking vinyl monomer which has two organic functions, three organic functions, and four organic functions in this copolymer chain attracts attention comparatively. That is, the acrylamide system copolymer of said structure is supposed that the paper durability effectiveness excellent in large pH field is acquired, and many proposals are made about this point. (For example, JP,63-50597,A, JP,2-61197,A, JP,4-18190,A, JP,3-227482,A, the special price **** No. 227483 [three to] official report, JP,5-14089,A, etc.)

[0005] However, in the vinyl system monomer which constitutes said copolymer, if there are too many additions of a cross-linking vinyl monomer, gelation will take place locally, and it has the fault from which copolymerization serves as water-insoluble nature. Actually, in order to solve said fault, the amount of the cross-linking vinyl monomer used is restricted to less than [1 mol %]. For this reason, since installation of the branching structure of playing an important role when giving the property excellent in quality of paper is restricted, by carrying out little addition

of the reactive surface active agent in JP,2-8207,A, local gelation was prevented and the approach of obtaining a uniform water solution has been proposed. Moreover, by using specific N-permutation (meta) acrylamide in JP,7-9779,A, introducing much branching structures into a polymer is proposed. However, this approach is also still insufficient, in order to solve said fault. [0006]

[The technical problem which is going to solve a problem] As everyone knows, it sets like the paper maker of paper and the paper board, and energy saving and saving-resources-ization are strongly called for from the environmental problem as mentioned above, for this reason, as for the raw material of paper, it is alike. The demand of upgrading accompanying the improvement corresponding to severe paper-making conditions, such as improvement in the speed of the paper-making machine for raising yield aggravation of the microfilament by the increment in the rate of combination of used paper and closing-izing of the paper-making process by wastewater regulation, a loading material, an internal chemical, etc. and productivity and deterioration of quality, or diversification of paper etc. is growing increasingly. However, the conventional additive for paper manufacture was still inadequate, in order to solve these problems. That is, the additive for paper manufacture which discovers the outstanding filterability, the yield, and a paper durability enhancing effect is demanded strongly.

[0007] In order that this invention persons may solve said many problems, as a result of inquiring wholeheartedly that the fault of the conventional technique should be canceled, it found out discovering the filterability excellent in the additive for paper manufacture with which a first-class alkenyl amine and/or its salts contain both the ionicity acrylamide system copolymer that has branching / bridge formation capacity and uses this first-class alkenyl amine and/or its salts as an indispensable component, the yield, and a paper durability enhancing effect.

[0008]

[Means for Solving the Problem] If this invention is outlined, it will be an additive for paper manufacture containing both the ionicity acrylamide system copolymer that uses the 1st class alkenyl amine and/or its salts as an indispensable component the first of this invention. Moreover, the second of this invention is the paper manufacture approach which adds and carries out paper making of the additive for paper manufacture of said this invention to a pulp slurry.

[0009] This invention is the new additive for paper manufacture and the paper manufacture approach the additive for paper manufacture containing both the ionicity acrylamide system copolymer that uses the 1st class alkenyl amine and/or its salts as an indispensable component discovers the filterability which was excellent in broad pH field also in severe paper-making conditions, the yield, and a paper durability enhancing effect, as described above. That is, this invention is both ionicity acrylamide system copolymer obtained by carrying out the polymerization of the vinyl monomer mixture which comes to contain the 1st class alkenyl amine and/or its salts (a), acrylamides (b), an anionic vinyl monomer (c), and a cationic vinyl monomer (d). furthermore -- desirable -- one -- class -- the alkenyl -- an amine -- and/or -- the -- salts -- (-- a --) -- 0.01 - 20 -- a mol -- % -- acrylamide -- a kind -- (-- b --) -- 40 - 98.99 -- a mol -- % -- anionic -- vinyl -- a monomer -- (-- c --) -- 0.5 - 20 -- a mol -- % -- cationicity -- vinyl -- a monomer -- (-- d --) -- 0.5 - 20 -- a mol -- % -- containing -- becoming -- vinyl -- a monomer -- mixture -- a polymerization -- carrying out -- obtaining -- having -- both -- ionicity -- acrylamide -- a system -- a copolymer -- it is -- paper manufacture -- ** -- an additive -- it is .

[0010] As the alkenyl amine used for both the ionicity acrylamide system copolymer in this invention, and/or its salts (a) A 2-propene-1-amine, a 2-methyl-2-propene-1-amine, These hydrochloric acids, such as a 2-ethyl-2-propene-1-amine, or a sulfuric acid, The salts of inorganic acids, such as phosphoric acid, sulfamic acid, an acetic acid, a propionic acid, a lactic acid, a citric acid, a malic acid, and a succinic acid, or an organic acid may be mentioned, and these kinds may be used independently, or two or more sorts may be used together.

[0011] As acrylamides (b) used for both the ionicity acrylamide system copolymer in this invention For example, acrylamide (meta), N-methyl (meta) acrylamide, N-ethyl (meta) acrylamide, N-isopropyl (meta) acrylamide, N-butyl (meta) acrylamide, N, and N-dimethyl (meta)

acrylamide, N, and N-diethyl (meta) acrylamide, diacetone (meta) acrylamide, etc. are mentioned, and these kinds may be used independently and may be used together two or more sorts. It is desirable to use acrylamide as a principal component from the field of cost performance.

[0012] As an anionic vinyl monomer (c) used for both the ionicity acrylamide system copolymer in this invention, unsaturated carboxylic acid, such as an acrylic acid, a methacrylic acid, a crotonic acid, a vinyl acetic acid, an itaconic acid, a maleic acid, a fumaric acid, muconic acid, a citraconic acid, and mesaconic acid, and these alkali-metal salts, an alkaline-earth-metal salt, and ammonium salt may be mentioned, these kinds may be used independently, for example, and two or more sorts may be used together.

[0013] As a cationic vinyl monomer (d) used for both the ionicity acrylamide system copolymer in this invention For example, diethylaminoethyl (meta) acrylate, diethylaminoethyl (meta) acrylate, An acrylic ester derivative like dimethylaminopropyl (meta) acrylate and diethylamino propyl (meta) acrylate (meta), An inorganic acid thru/or organic acids, such as acrylamide derivatives like dimethylaminopropyl (meta) acrylamide (meta) or these hydrochloric acids, a sulfuric acid, a nitric acid, phosphoric acid, sulfamic acid, an acetic acid, a propionic acid, a lactic acid, a citric acid, a malic acid, and a succinic acid, can be illustrated. Moreover, the quarternary-ammonium-salt vinyl monomer which turned the 3rd class amino group of these vinyl monomers the fourth class with alkyl halide, phenyl alkyl halide, a dialkyl sulfuric acid, epichlorohydrin, a chloroacetic acid, etc., for example, acryloyloxyethyl (meta) trimethylammonium chloride, acryloyloxyethyl (meta) dimethylbenzyl ammoniumchloride, acryloyloxyethyl (meta) trimethylammonium methyl sulfate, etc. can be illustrated. Moreover, in addition to this, diaryl dimethylammonium chloride, 3-(meth)acryloyloxy 2-hydroxypropyl trimethylammoniumchloride, etc. can be illustrated. And these kinds may be used independently and two or more sorts may be used together.

[0014] In this invention, it is the disadvantage **** range substantially about the effectiveness of this invention other than said configuration vinyl monomer, and said configuration vinyl monomer and the vinyl monomer which can be copolymerized can be used. As a vinyl monomer in which this kind of copolymerization is possible Acrylic nitril, vinyl acetate, styrene, alpha methyl styrene, and acrylic-acid (meta) alkyl ester The Nonion nature vinyl monomers, such as N-vinyl pyrrolidone, N-vinyl formamide, and N-vinyl caprolactam, Glycidyl (meta) acrylate, methylol (meta) acrylamide, Reactant monomers, such as methoxymethyl (meta) acrylamide, methylenebis (meta) acrylamide, Ethylene screw (meta) acrylamide, a divinylbenzene, an adipic-acid divinyl, Polyfunctional vinyl monomers, such as ethylene GURIKORUJI (meta) acrylate, 1 and 3, 5-thoria chestnut roil hexahydro-1,3,5-triazine, and triallyl isocyanurate, etc. are mentioned.

[0015] In both the ionicity acrylamide system copolymer of this invention, the (d) component 0.5 - the 20-mol range of the mol rate of (a) - (d) component are % the (c) component and 0.5 - 20-mol% (b) component 40 - 98.99-mol% (a) component 0.01 - 20-mol%. It is (b) component 70 97.9-mol % (c) component 1 ten-mol % (d) component 1 - ten-mol % (a) component 0.1 - ten-mol% preferably. In addition, when the polymer which has sufficient branching structure when (a) is less than [0.01 mol %] is not obtained but it exceeds 20-mol %, the copolymer obtained gels, it becomes water-insoluble nature, and the additive for paper manufacture of this invention is not obtained. Moreover, when the rate of (b), (c), and the (d) component is outside the above-mentioned range, the purpose of this invention is not reached.

[0016] In order to manufacture the additive for paper manufacture concerning this invention, the above (a), (b), (c), and the (d) component take into consideration the class of paper to manufacture, paper-making conditions, etc., and are used at optimal operating rate.

[0017] Manufacture of the additive for paper manufacture concerning this invention can be performed by the approach better known than before. For example, by teaching a chain transfer agent to a reaction container a (a) component - (d) component, water, and if needed so that it may usually become 5 - 30% of the weight, and adding and warming the bottom radical polymerization initiator of stirring, the monomer concentration at the time of preparation performs a polymerization, and obtains the additive for paper manufacture concerning this invention. Any of a batch reaction and a half-batch reaction are sufficient as the gestalt of a polymerization reaction. Reaction temperature is 50-90 degrees C, and reaction time is usually

about 1 – 5 hours. As a radical polymerization initiator, azo compounds, such as peroxides [, such as persulfate, such as ammonium persulfate and potassium persulfate, a hydrogen peroxide and a benzoyl peroxide,], 2, and 2-azo hysteries (2-amidinopropane) hydrochloride and azobisisobutyronitril, etc. can be illustrated here, these kinds may be used independently, and two or more sorts may be used together. When using the matter of oxidizing qualities, such as persulfate and *****, for an initiator, it can use together with a reducing agent and redox polymerization can be performed. A sulfite like a sodium sulfite as a reducing agent, a hydrogensulfite like a sodium hydrogensulfite, etc. are mentioned here. The chain transfer agent used here is well-known, and can illustrate phosphinate, such as allyl compounds (meta), such as good for example, thiols like a mercaptan, thioglycolic acid and its ester, allyl alcohol, sodium allylsulfonate, and sodium methallylsulfonate, alcohols like isopropyl alcohol, and phosphinic acid sodium, etc. Moreover, in order to decrease a residual monomer, adding a polymerization initiator over several order does not interfere, either.

[0018] Generally the additive for paper manufacture concerning this invention is added in paper-making processes, such as paper and the paper board, by the wet end sections in a pulp slurry, on a wet paper web, etc. As the rate of addition, it is 0.03 – 3 % of the weight preferably 0.01 to 10% of the weight in solid content to pulp bone-dry solid content. The pulp slurry at the time of addition or pH of a wet paper web is 4–8. As a class of pulp, non-cellulose fiber, such as various kinds of pulp, such as chemical pulp, mechanical pulp, corrugated paper recycled pulp, magazine recycled pulp, or newspaper recycled pulp, or glass wool, and a chemical fiber, can be mixed and used. In addition, a color, a content, a loading material, pH modifiers, such as a sulfuric acid and a sodium hydroxide, a sizing compound, a humid paper durability improver, a desiccation paper reinforcing agent, a filtered water improver, a yield improver, a defoaming agent, or a slime control agent can be used together if needed. Moreover, it can be used for acid paper making from all neutral paper making, for example, can be used for a corrugated paper forms [, such as paper of fine quality, a report grade paper, coat paper, a newsprint, a converting paper a liner and green sand core stencil paper,], PPC form, inkjet-printing-paper, thermal paper, special paper, and toner printer form, a plaster board form, a building-materials form, etc.

[0019]

[Embodiment of the Invention] The gestalt of operation of this invention is as follows.

[0020] In the reaction flask equipped with [preparation of additive for paper manufacture] thermometer, an agitator, a cooling pipe, and nitrogen gas installation tubing, a 2-propene-1-amine, Vinyl monomer components, such as 50% of acrylamide solution, dimethylaminoethyl methacrylate, and an itaconic acid, Sodium allylsulfonate and water are prepared as a chain transfer agent of the specified quantity. It prepares to pH in a sulfuric-acid water solution 20%, a temperature up is carried out to 55 degrees C under nitrogen gas installation, then, specified quantity addition of an ammonium persulfate water solution and the 5% sodium-hydrogensulfite water solution is carried out 50% as a polymerization initiator, a polymerization reaction is carried out at 80 degrees C for 2 hours, and both the ionicity acrylamide system copolymer is obtained. [0021] [-- quality of paper -- 1.5% of solid content for pulp was added for the sulfuric-acid band to the corrugated paper used paper (C. S.F370ml) pulp slurry of 3% of evaluation approach] concentration, and it prepared to pH5.8. Next, said both ionicity acrylamide system copolymer is added 0.5% of solid content for pulp, paper equivalent to basis-weight 150 g/m² is dried for the obtained pulp slurry for 3 minutes in 5 minutes and on a 110-degree C hot plate by **** and 5kg/cm² of press ** by the TAPPI standard sheet machine, and the test paper is created. the following quality of paper after carrying out gas conditioning of the test paper for 24 hours to the bottom of the condition of the temperature of 20 degrees C, and 65% of humidity -- it examines by the appraisal method.

[0022] (1) quality of paper -- evaluation approach compressive strength: -- JIS Bursting strength:JIS according to P8126 Tensile strength:JIS according to P8112 It applies to P8113 correspondingly. [0023] a [filterability test method] -- said quality of paper -- the water which prepared the corrugated paper recycled pulp slurry of 3% of concentration which added the chemical like the paper-making conditions in the evaluation approach, and prepared it to the same pH as a pulp slurry -- 0.3% -- diluting -- the 100ml -- using -- JIS According to P8121, it

measured with the Canadian freeness circuit tester.

[0024] The corrugated paper recycled pulp slurry of 3% of concentration which added the chemical like the paper-making conditions in the evaluation approach is prepared. the [yield effectiveness test method] -- said quality of paper -- It is diluted with the water prepared to the same pH as a pulp slurry to 0.6%. The 500ml TAPPI JOURNAL 56 (10) dynamic given in 46-50 (1973) drainage PAPER of the same equipment as jar RESEARCH MATERIALS, Britt made from INC Jar It flows into Tester. After stirring by 700rpm during 20 seconds, it drains for 10 seconds, and 100ml of filtrate is extracted continuously. The filtrate was filtered through the filter paper of No.3, and the value of a first pass retention was calculated from the amount of the residue.

[0025]

[Example] Hereafter, an example and the example of a comparison are shown and this invention is explained more to a detail. In addition, each % is weight percent as long as there is no assignment especially.

[0026] It is 162g (91.5-mol%) of 50% acrylamide water solutions to the 500ml four-lot flask equipped with a [example 1] thermometer, an agitator, a cooling pipe, and nitrogen gas installation tubing, Dimethylamino methyl methacrylate 9.80g (five-mol%), 4.06g (2.5-mol%) of itaconic acids, 2-propene-1-amine 0.721g (one-mol%), 18g of 3% sodium methallylsulfonate water solutions, Prepare 310g of water, and 13.8g of 20% sulfuric-acid water solutions, and pH is prepared to 4.0. Carried out the temperature up to 55 degrees C under nitrogen gas installation, subsequently added 2.88ml of ammonium persulfate water solutions, and 0.72ml of 5% sodium-hydrogensulfite water solutions 5%, it was made to react at 80 degrees C for 2 hours, and both the ionicity acrylamide system copolymer A was obtained. The description of the polymer is shown in Table 1.

[0027] In the [examples 2-7] example 1, both ionicity acrylamide system copolymer B-G was obtained by the same approach as an example except having changed the class of vinyl monomer component, or its monomer presentation (mol %), as shown in Table 1. The description of the copolymer is shown in Table 1.

[0028] In the [examples 1-5 of comparison] example 1, acrylamide system copolymer H-M was obtained by the same approach as an example 1 except having changed the class of vinyl monomer component, or its monomer presentation (mol %), as shown in Table 1. The description is shown in Table 1.

[0029] In addition, the semantics of the cable address used in Table 1 is as follows.

AAM: -- acrylamide -- MAAM: methacrylamide DMAA: dimethyl acrylamide DM: dimethylaminoethyl methacrylate DMB: -- methacryloiloxy-ethyl -- dimethylbenzyl ammoniumchloride DMC: methacryloiloxy-ethyl trimethylammonium chloride IA: itaconic-acid AA: -- an acrylic acid AAn -- :2-propene-1-amine MBA: methylenebis acrylamide TAF: -- 1, 3, and 5-thoria KUROIU hexahydro-1,3,5-triazine MMA: methyl methacrylate St: styrene [0030]

[Table 1]

		共重合体	各成分のモル%					固形分 %	粘度 cps/25℃	pH
			(a)	(b)	(c)	(d)	他のモノマー			
実施例	1	A	AAn 1	AAm 91.5	IA 2.5	DM 5	—	20.5	7540	4.3
	2	B	AAn 5	AAm 87.5	IA 2.5	DM 2.5 DMB 2.5	—	20.5	8260	4.2
	3	C	AAn 3	AAm 89.5	IA 2.5	DMB 5	TAF 0.01	20.6	8120	4.3
	4	D	AAn 5	AAm 85	AA 5	DM 2.5 DMC 2.5		20.5	8360	3.8
	5	E	AAn 2	AAm 88	IA 2.5	DMAPAA 5	St 2.5	20.6	8020	4.4
	6	F	AAn 3	AAm 84.5 DMAA 2.5	AA 5	DM 2.5 DMB 2.5		20.8	7780	3.9
	7	G	AAn 2	AAm 81	AA 5	DM 10	MMA 2			
比較例	1	H	—	AAm 92.5	IA 2.5	DM 5	—	20.4	8750	4.4
	2	I	—	AAm 89.99	AA 5	DM 2.5 DMB 2.5	TAF 0.01	20.4	8650	3.9
	3	J	—	AAm 92.4	IA 2.5	DMAPAA 5	MBA 0.1	20.3	8220	4.3
	4	K		AAm 89.8 DMAA 2.5	IA 2.5	DM 2.5 DMB 2.5	MBA 0.2	20.4	8170	4.4
	5	M	AAn 2	AAm 93		DMAPAA 5	—	20.4	8310	4.2

[0031] applications 1-7 and [comparison application 1-5] corrugated paper used paper -- a Niagara style -- beating was carried out with the beater, and the sulfuric-acid band was added 1.5 % of the weight of opposite pulp to 3% of the weight of the recycled pulp slurry prepared to Canadian standard freeness (C. S.F) 370ml, and it was referred to as pH5.8. the copolymer obtained in each above-mentioned examples 1-7 and the example of a comparison -- solid content -- 0.5wt% for pulp -- it added, and by the TAPPI standard sheet machine, paper equivalent to basis-weight 150 g/m2 was pressed for 5 minutes by **** and press ** 5 kg/m2, the obtained pulp slurry liquid was dried for 3 minutes on the 110 more-degree C hot plate, and the test paper was created. Furthermore, JIS after carrying out gas conditioning under conditions of the temperature of 20 degrees C, and 65% of humidity for 24 hours According to P8126, P8112, and P8113, compressive strength, bursting strength, and tensile strength were measured. The result is shown in Table 2. In addition, measurement of filterability and the yield effectiveness was performed according to the following. The result is shown in Table 2.

[0032] It dilutes with the tap water which prepared the paper pulp slurry for corrugated paper of 3% of concentration which added the chemical like the paper-making conditions in the [filterability trial] applications 1-7 and the examples 1-5 of a comparison, and prepared it to the same pH as a pulp slurry to 0.3%, the 100ml is used, and it is JIS. According to P8121, it measured with the Canadian freeness circuit tester.

[0033] It is a chemical like the paper-making conditions in the [yield effectiveness trial] applications 1-7 and the examples 1-5 of a comparison. It dilutes with the water which prepared the corrugated paper recycled pulp slurry of 3% of added concentration, and prepared it to the same pH as a pulp slurry to 0.6%. The 500ml TAPPI JOURNAL 56 (10) dynamicdrainage given in 46-50 (OCT.1973) PAPER of the same equipment as Jar RESEARCH MATERIALS and Britt made from INC Jar After flowing into Tester and stirring by 700rpm during 20 seconds, it drains for 10 seconds, and 100ml of filtrate is extracted continuously. The filtrate was filtered through

the filter paper of No.3, and the value of a first pass retention was calculated from the amount of the residue.

[0034]

[Table 2]

		共 重 合 体	比圧縮強度	比破裂強度	裂 断 長 km	C. S. F	OPR %
応 用 例	1	A	18. 0	2. 63	4. 56	390	80. 7
	2	B	18. 5	2. 68	4. 62	410	81. 8
	3	C	18. 7	2. 71	4. 77	400	81. 7
	4	D	18. 1	2. 65	4. 58	400	81. 3
	5	E	18. 0	2. 62	4. 56	390	80. 8
	6	F	18. 2	2. 75	4. 70	400	82. 2
	7	G	18. 3	2. 75	4. 65	395	81. 2
応 用 比 較 例	1	H	17. 3	2. 44	4. 25	380	79. 0
	2	I	17. 6	2. 51	4. 36	385	80. 1
	3	J	17. 4	2. 57	4. 22	380	79. 4
	4	K	17. 5	2. 58	4. 38	385	79. 9
	5	M	16. 6	2. 24	4. 01	380	79. 3

[0035]

[Effect of the Invention] It is clear that the additive's for paper manufacture which consists the first-class alkenyl amine of this invention and/or its salts of both ionicity acrylamide system copolymer prepared as an indispensable constituent the effectiveness which was excellent in large pH field in filtered water, the yield, and a paper durability enhancing effect is shown as compared with the conventional additive for paper manufacture a passage clear from the result shown in Table 2.

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TECHNICAL FIELD

[Field of the Invention] This invention relates to the additive for paper manufacture and the paper manufacture approach of consisting of both ionicity acrylamide system copolymer set and used like a paper maker. Furthermore, it is related with the new additive for paper manufacture and the paper manufacture approach of discovering the filterability which was excellent in detail also in the paper-making conditions in neutral thru/or alkaline broad pH field from acidity, the yield, and a paper durability enhancing effect.

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PRIOR ART

[Description of the Prior Art] the increment in the used paper utilization factor set like the paper maker of paper and the paper board, and according to factors, such as aggravation of a bolt supply situation, and energy saving, and closing-izing of a paper-making system -- progressing -- quality of paper -- when planning improvement and a productivity drive, the importance of a paper reinforcing agent, a filtration improvement agent, and a yield improver increases, and these various additives for paper manufacture are offered.

[0003] As an additive for paper manufacture, starch, a polyamide polyamine epichlorohydrin resin, polyacrylamide system resin, etc. are used widely, and use of polyacrylamide system resin is expanded especially. As a class of additive for paper manufacture which consists of polyacrylamide system resin, there are an anionic polymer, a cationic polymer, and both ionicity polymer greatly from the difference in the ionicity. Also under the paper-making conditions that both the ionicity copolymer obtained by aggravation of the paper-making raw material of this inside or recent years, improvement in a paper-making rate, etc. by copolymerizing an anionic vinyl monomer and a cationic vinyl monomer with acrylamide (meta) is severe, since there are comparatively few falls of effectiveness, the rate of an use rate has increased. However, these linear types of polymer is also inadequate for corresponding to a demand of high quality which was described above, aggravation of paper-making conditions, etc.

[0004] It faces carrying out the polymerization of acrylamide (meta), a cationic vinyl monomer, and the anionic vinyl monomer, and preparing an acrylamide system copolymer recently, and the acrylamide system copolymer which has the high branching structure of the amount of macromolecules which introduced the cross-linking vinyl monomer which has two organic functions, three organic functions, and four organic functions in this copolymer chain attracts attention comparatively. That is, the acrylamide system copolymer of said structure is supposed that the paper durability effectiveness excellent in large pH field is acquired, and many proposals are made about this point. (For example, JP,63-50597,A, JP,2-61197,A, JP,4-18190,A, JP,3-227482,A, the special price **** No. 227483 [three to] official report, JP,5-14089,A, etc.)

[0005] However, in the vinyl system monomer which constitutes said copolymer, if there are too many additions of a cross-linking vinyl monomer, gelation will take place locally, and it has the fault from which copolymerization serves as water-insoluble nature. Actually, in order to solve said fault, the amount of the cross-linking vinyl monomer used is restricted to less than [1 mol %]. For this reason, since installation of the branching structure of playing an important role when giving the property excellent in quality of paper is restricted, by carrying out little addition of the reactive surface active agent in JP,2-8207,A, local gelation was prevented and the approach of obtaining a uniform water solution has been proposed. Moreover, by using specific N-permutation (meta) acrylamide in JP,7-9779,A, introducing much branching structures into a polymer is proposed. However, this approach is also still insufficient, in order to solve said fault.

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EFFECT OF THE INVENTION

[Effect of the Invention] It is clear that the additive's for paper manufacture which consists the first-class alkenyl amine of this invention and/or its salts of both ionicity acrylamide system copolymer prepared as an indispensable constituent the effectiveness which was excellent in large pH field in filtered water, the yield, and a paper durability enhancing effect is shown as compared with the conventional additive for paper manufacture a passage clear from the result shown in Table 2.

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TECHNICAL PROBLEM

[The technical problem which is going to solve a problem] As everyone knows, it sets like the paper maker of paper and the paper board, and energy saving and saving-resources-ization are strongly called for from the environmental problem as mentioned above, for this reason, as for the raw material of paper, it is alike. The demand of upgrading accompanying the improvement corresponding to severe paper-making conditions, such as improvement in the speed of the paper-making machine for raising yield aggravation of the microfilament by the increment in the rate of combination of used paper and closing-izing of the paper-making process by wastewater regulation, a loading material, an internal chemical, etc. and productivity and deterioration of quality, or diversification of paper etc. is growing increasingly. However, the conventional additive for paper manufacture was still inadequate, in order to solve these problems. That is, the additive for paper manufacture which discovers the outstanding filterability, the yield, and a paper durability enhancing effect is demanded strongly.

[0007] In order that this invention persons may solve said many problems, as a result of inquiring wholeheartedly that the fault of the conventional technique should be canceled, it found out discovering the filterability excellent in the additive for paper manufacture with which a first-class alkenyl amine and/or its salts contain both the ionicity acrylamide system copolymer that has branching / bridge formation capacity and uses this first-class alkenyl amine and/or its salts as an indispensable component, the yield, and a paper durability enhancing effect.

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MEANS

[Means for Solving the Problem] If this invention is outlined, it will be an additive for paper manufacture containing both the ionicity acrylamide system copolymer that uses the 1st class alkenyl amine and/or its salts as an indispensable component the first of this invention. Moreover, the second of this invention is the paper manufacture approach which adds and carries out paper making of the additive for paper manufacture of said this invention to a pulp slurry.

[0009] This invention is the new additive for paper manufacture and the paper manufacture approach the additive for paper manufacture containing both the ionicity acrylamide system copolymer that uses the 1st class alkenyl amine and/or its salts as an indispensable component discovers the filterability which was excellent in broad pH field also in severe paper-making conditions, the yield, and a paper durability enhancing effect, as described above. That is, this invention is both ionicity acrylamide system copolymer obtained by carrying out the polymerization of the vinyl monomer mixture which comes to contain the 1st class alkenyl amine and/or its salts (a), acrylamides (b), an anionic vinyl monomer (c), and a cationic vinyl monomer (d). furthermore -- desirable -- one -- class -- the alkenyl -- an amine -- and/or -- the -- salts -- (-- a --) -- 0.01 - 20 -- a mol -- % -- acrylamide -- a kind -- (-- b --) -- 40 - 98.99 -- a mol -- % -- anionic -- vinyl -- a monomer -- (-- c --) -- 0.5 - 20 -- a mol -- % -- cationicity -- vinyl -- a monomer -- (-- d --) -- 0.5 - 20 -- a mol -- % -- containing -- becoming -- vinyl -- a monomer -- mixture -- a polymerization -- carrying out -- obtaining -- having -- both -- ionicity -- acrylamide -- a system -- a copolymer -- it is -- paper manufacture -- ** -- an additive -- it is .

[0010] As the alkenyl amine used for both the ionicity acrylamide system copolymer in this invention, and/or its salts (a) A 2-propene-1-amine, a 2-methyl-2-propene-1-amine, These hydrochloric acids, such as a 2-ethyl-2-propene-1-amine, or a sulfuric acid, The salts of inorganic acids, such as phosphoric acid, sulfamic acid, an acetic acid, a propionic acid, a lactic acid, a citric acid, a malic acid, and a succinic acid, or an organic acid may be mentioned, and these kinds may be used independently, or two or more sorts may be used together.

[0011] As acrylamides (b) used for both the ionicity acrylamide system copolymer in this invention For example, acrylamide (meta), N-methyl (meta) acrylamide, N-ethyl (meta) acrylamide, N-isopropyl (meta) acrylamide, N-butyl (meta) acrylamide, N, and N-dimethyl (meta) acrylamide, N, and N-diethyl (meta) acrylamide, diacetone (meta) acrylamide, etc. are mentioned, and these kinds may be used independently and may be used together two or more sorts. It is desirable to use acrylamide as a principal component from the field of cost performance.

[0012] As an anionic vinyl monomer (c) used for both the ionicity acrylamide system copolymer in this invention, unsaturated carboxylic acid, such as an acrylic acid, a methacrylic acid, a crotonic acid, a vinyl acetic acid, an itaconic acid, a maleic acid, a fumaric acid, muconic acid, a citraconic acid, and mesaconic acid, and these alkali-metal salts, an alkaline-earth-metal salt, and ammonium salt may be mentioned, these kinds may be used independently, for example, and two or more sorts may be used together.

[0013] As a cationic vinyl monomer (d) used for both the ionicity acrylamide system copolymer in this invention For example, diethylaminoethyl (meta) acrylate, diethylaminoethyl (meta)

acrylate, An acrylic ester derivative like dimethylaminopropyl (meta) acrylate and diethylamino propyl (meta) acrylate (meta), An inorganic acid thru/or organic acids, such as acrylamide derivatives like dimethylaminopropyl (meta) acrylamide (meta) or these hydrochloric acids, a sulfuric acid, a nitric acid, phosphoric acid, sulfamic acid, an acetic acid, a propionic acid, a lactic acid, a citric acid, a malic acid, and a succinic acid, can be illustrated. Moreover, the quarternary-ammonium-salt vinyl monomer which turned the 3rd class amino group of these vinyl monomers the fourth class with alkyl halide, phenyl alkyl halide, a dialkyl sulfuric acid, epichlorohydrin, a chloroacetic acid, etc., for example, acryloyloxyethyl (meta) trimethylammonium chloride, acryloyloxyethyl (meta) dimethylbenzyl ammoniumchloride, acryloyloxyethyl (meta) trimethylammonium methyl sulfate, etc. can be illustrated. Moreover, in addition to this, diaryl dimethylammonium chloride, 3-(meth)acryloyloxy 2-hydroxypropyl trimethylammoniumchloride, etc. can be illustrated. And these kinds may be used independently and two or more sorts may be used together.

[0014] In this invention, it is the disadvantage **** range substantially about the effectiveness of this invention other than said configuration vinyl monomer, and said configuration vinyl monomer and the vinyl monomer which can be copolymerized can be used. As a vinyl monomer in which this kind of copolymerization is possible Acrylic nitril, vinyl acetate, styrene, alpha methyl styrene, and acrylic-acid (meta) alkyl ester The Nonion nature vinyl monomers, such as N-vinyl pyrrolidone, N-vinyl formamide, and N-vinyl caprolactam, Glycidyl (meta) acrylate, methylol (meta) acrylamide, Reactant monomers, such as methoxymethyl (meta) acrylamide, methylenebis (meta) acrylamide, Ethylene screw (meta) acrylamide, a divinylbenzene, an adipic-acid divinyl, Polyfunctional vinyl monomers, such as ethylene GURIKORUJI (meta) acrylate, 1 and 3, 5-thoria chestnut roil hexahydro-1,3,5-triazine, and triallyl isocyanurate, etc. are mentioned.

[0015] In both the ionicity acrylamide system copolymer of this invention, the (d) component 0.5 - the 20-mol range of the mol rate of (a) - (d) component are % the (c) component and 0.5 - 20-mol% (b) component 40 - 98.99-mol% (a) component 0.01 - 20-mol%. It is (b) component 70 97.9-mol % (c) component 1 ten-mol % (d) component 1 - ten-mol % (a) component 0.1 - ten-mol% preferably. In addition, when the polymer which has sufficient branching structure when (a) is less than [0.01 mol %] is not obtained but it exceeds 20-mol %, the copolymer obtained gels, it becomes water-insoluble nature, and the additive for paper manufacture of this invention is not obtained. Moreover, when the rate of (b), (c), and the (d) component is outside the above-mentioned range, the purpose of this invention is not reached.

[0016] In order to manufacture the additive for paper manufacture concerning this invention, the above (a), (b), (c), and the (d) component take into consideration the class of paper to manufacture, paper-making conditions, etc., and are used at optimal operating rate.

[0017] Manufacture of the additive for paper manufacture concerning this invention can be performed by the approach better known than before. For example, by teaching a chain transfer agent to a reaction container a (a) component - (d) component, water, and if needed so that it may usually become 5 - 30% of the weight, and adding and warming the bottom radical polymerization initiator of stirring, the monomer concentration at the time of preparation performs a polymerization, and obtains the additive for paper manufacture concerning this invention. Any of a batch reaction and a half-batch reaction are sufficient as the gestalt of a polymerization reaction. Reaction temperature is 50-90 degrees C, and reaction time is usually about 1 - 5 hours. As a radical polymerization initiator, azo compounds, such as peroxides [, such as persulfate, such as ammonium persulfate and potassium persulfate, a hydrogen peroxide and a benzoyl peroxide,], 2, and 2-azo hysterics (2-amidinopropane) hydrochloride and azobisisobutyronitril, etc. can be illustrated here, these kinds may be used independently, and two or more sorts may be used together. When using the matter of oxidizing qualities, such as persulfate and *****, for an initiator, it can use together with a reducing agent and redox polymerization can be performed. A sulfite like a sodium sulfite as a reducing agent, a hydrogensulfite like a sodium hydrogensulfite, etc. are mentioned here. The chain transfer agent used here is well-known, and can illustrate phosphinate, such as allyl compounds (meta), such as good for example, thiols like a mercaptan, thioglycolic acid and its ester, allyl alcohol, sodium allylsulfonate, and sodium methallylsulfonate, alcohols like isopropyl alcohol, and phosphinic acid

sodium, etc. Moreover, in order to decrease a residual monomer, adding a polymerization initiator over several order does not interfere, either.

[0018] Generally the additive for paper manufacture concerning this invention is added in paper-making processes, such as paper and the paper board, by the wet end sections in a pulp slurry, on a wet paper web, etc. As the rate of addition, it is 0.03 – 3 % of the weight preferably 0.01 to 10% of the weight in solid content to pulp bone-dry solid content. The pulp slurry at the time of addition or pH of a wet paper web is 4–8. As a class of pulp, non-cellulose fiber, such as various kinds of pulp, such as chemical pulp, mechanical pulp, corrugated paper recycled pulp, magazine recycled pulp, or newspaper recycled pulp, or glass wool, and a chemical fiber, can be mixed and used. In addition, a color, a content, a loading material, pH modifiers, such as a sulfuric acid and a sodium hydroxide, a sizing compound, a humid paper durability improver, a desiccation paper reinforcing agent, a filtered water improver, a yield improver, a defoaming agent, or a slime control agent can be used together if needed. Moreover, it can be used for acid paper making from all neutral paper making, for example, can be used for a corrugated paper forms [, such as paper of fine quality, a report grade paper, coat paper, a newsprint, a converting paper a liner and green sand core stencil paper,], PPC form, inkjet-printing-paper, thermal paper, special paper, and toner printer form, a plaster board form, a building-materials form, etc.

[0019]

[Embodiment of the Invention] The gestalt of operation of this invention is as follows.

[0020] In the reaction flask equipped with [preparation of additive for paper manufacture] thermometer, an agitator, a cooling pipe, and nitrogen gas installation tubing, a 2-propene-1-amine, Vinyl monomer components, such as 50% of acrylamide solution, dimethylaminoethyl methacrylate, and an itaconic acid, Sodium allylsulfonate and water are prepared as a chain transfer agent of the specified quantity. It prepares to pH in a sulfuric-acid water solution 20%, a temperature up is carried out to 55 degrees C under nitrogen gas installation, then, specified quantity addition of an ammonium persulfate water solution and the 5% sodium-hydrogensulfite water solution is carried out 50% as a polymerization initiator, a polymerization reaction is carried out at 80 degrees C for 2 hours, and both the ionicity acrylamide system copolymer is obtained.

[0021] [— quality of paper — 1.5% of solid content for pulp was added for the sulfuric-acid band to the corrugated paper used paper (C. S.F370ml) pulp slurry of 3% of evaluation approach] concentration, and it prepared to pH5.8. Next, said both ionicity acrylamide system copolymer is added 0.5% of solid content for pulp, paper equivalent to basis-weight 150 g/m² is dried for the obtained pulp slurry for 3 minutes in 5 minutes and on a 110-degree C hot plate by **** and 5kg/cm² of press ** by the TAPPI standard sheet machine, and the test paper is created. the following quality of paper after carrying out gas conditioning of the test paper for 24 hours to the bottom of the condition of the temperature of 20 degrees C, and 65% of humidity — it examines by the appraisal method.

[0022] (1) quality of paper — evaluation approach compressive strength: — JIS Bursting strength:JIS according to P8126 Tensile strength:JIS according to P8112 It applies to P8113 correspondingly. [0023] a [filterability test method] — said quality of paper — the water which prepared the corrugated paper recycled pulp slurry of 3% of concentration which added the chemical like the paper-making conditions in the evaluation approach, and prepared it to the same pH as a pulp slurry — 0.3% — diluting — the 100ml — using — JIS According to P8121, it measured with the Canadian freeness circuit tester.

[0024] The corrugated paper recycled pulp slurry of 3% of concentration which added the chemical like the paper-making conditions in the evaluation approach is prepared. the [yield effectiveness test method] — said quality of paper — It is diluted with the water prepared to the same pH as a pulp slurry to 0.6%. The 500ml TAPPI JOURNAL 56 (10) dynamic given in 46–50 (1973) drainage PAPER of the same equipment as jar RESEARCH MATERIALS, Britt made from INC Jar It flows into Tester. After stirring by 700rpm during 20 seconds, it drains for 10 seconds, and 100ml of filtrate is extracted continuously. The filtrate was filtered through the filter paper of No.3, and the value of a first pass retention was calculated from the amount of the residue.

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EXAMPLE

[Example] Hereafter, an example and the example of a comparison are shown and this invention is explained more to a detail. In addition, each % is weight percent as long as there is no assignment especially.

[0026] It is 162g (91.5-mol%) of 50% acrylamide water solutions to the 500ml four-lot flask equipped with a [example 1] thermometer, an agitator, a cooling pipe, and nitrogen gas installation tubing, Dimethylamino methyl methacrylate 9.80g (five-mol%), 4.06g (2.5-mol%) of itaconic acids, 2-propene-1-amine 0.721g (one-mol%), 18g of 3% sodium methallylsulfonate water solutions, Prepare 310g of water, and 13.8g of 20% sulfuric-acid water solutions, and pH is prepared to 4.0. Carried out the temperature up to 55 degrees C under nitrogen gas installation, subsequently added 2.88ml of ammonium persulfate water solutions, and 0.72ml of 5% sodium-hydrogensulfite water solutions 5%, it was made to react at 80 degrees C for 2 hours, and both the ionicity acrylamide system copolymer A was obtained. The description of the polymer is shown in Table 1.

[0027] In the [examples 2-7] example 1, both ionicity acrylamide system copolymer B-G was obtained by the same approach as an example except having changed the class of vinyl monomer component, or its monomer presentation (mol %), as shown in Table 1. The description of the copolymer is shown in Table 1.

[0028] In the [examples 1-5 of comparison] example 1, acrylamide system copolymer H-M was obtained by the same approach as an example 1 except having changed the class of vinyl monomer component, or its monomer presentation (mol %), as shown in Table 1. The description is shown in Table 1.

[0029] In addition, the semantics of the cable address used in Table 1 is as follows.

AAM: -- acrylamide -- MAAM: methacrylamide DMAA: dimethyl acrylamide DM: dimethylaminoethyl methacrylate DMB: -- methacryloiloxy-ethyl -- dimethylbenzyl ammoniumchloride

DMC: methacryloiloxy-ethyl trimethylammonium chloride IA: itaconic-acid AA: -- an acrylic acid AAn -- :2-propene-1-amine MBA: methylenebis acrylamide TAF: -- 1, 3, and 5-thoria KUROIU hexahydro-1,3,5-triazine MMA: methyl methacrylate St: styrene [0030]

[Table 1]

	共重合体	各成分のモル%					固形分 %	粘度 cps/25℃	pH	
		(a)	(b)	(c)	(d)	他のビ ニルマ-				
実施例	1	A	AAn 1	AAm 91.5	IA 2.5	DM 5	—	20.5	7540	4.3
	2	B	AAn 5	AAm 87.5	IA 2.5	DM 2.5 DMB 2.5	—	20.5	8260	4.2
	3	C	AAn 3	AAm 89.5	IA 2.5	DMB 5	TAF 0.01	20.6	8120	4.3
	4	D	AAn 5	AAm 85	AA 5	DM 2.5 DMC 2.5		20.5	8360	3.8
	5	E	AAn 2	AAm 88	IA 2.5	DMAPAA 5	St 2.5	20.6	8020	4.4
	6	F	AAn 3	AAm 84.5 DMAA 2.5	AA 5	DM 2.5 DMB 2.5		20.3	7780	3.9
	7	G	AAn 2	AAm 81	AA 5	DM 10	MMA 2			
比較例	1	H	—	AAm 92.5	IA 2.5	DM 5	—	20.4	8750	4.4
	2	I	—	AAm 89.99	AA 5	DM 2.5 DMB 2.5	TAF 0.01	20.4	8650	3.9
	3	J	—	AAm 92.4	IA 2.5	DMAPAA 5	MBA 0.1	20.3	8220	4.3
	4	K		AAm 89.8 DMAA 2.5	IA 2.5	DM 2.5 DMB 2.5	MBA 0.2	20.4	8170	4.4
	5	M	AAn 2	AAm 93		DMAPAA 5	—	20.4	8310	4.2

[0031] applications 1-7 and [comparison application 1-5] corrugated paper used paper -- a Niagara style -- beating was carried out with the beater, and the sulfuric-acid band was added 1.5 % of the weight of opposite pulp to 3% of the weight of the recycled pulp slurry prepared to Canadian standard freeness (C. S.F) 370ml, and it was referred to as pH5.8. the copolymer obtained in each above-mentioned examples 1-7 and the example of a comparison -- solid content -- 0.5wt% for pulp -- it added, and by the TAPPI standard sheet machine, paper equivalent to basis-weight 150 g/m² was pressed for 5 minutes by **** and press ** 5 kg/m², the obtained pulp slurry liquid was dried for 3 minutes on the 110 more-degree C hot plate, and the test paper was created. Furthermore, JIS after carrying out gas conditioning under conditions of the temperature of 20 degrees C, and 65% of humidity for 24 hours According to P8126, P8112, and P8113, compressive strength, bursting strength, and tensile strength were measured. The result is shown in Table 2. In addition, measurement of filterability and the yield effectiveness was performed according to the following. The result is shown in Table 2.

[0032] It dilutes with the tap water which prepared the paper pulp slurry for corrugated paper of 3% of concentration which added the chemical like the paper-making conditions in the [filterability trial] applications 1-7 and the examples 1-5 of a comparison, and prepared it to the same pH as a pulp slurry to 0.3%, the 100ml is used, and it is JIS. According to P8121, it measured with the Canadian freeness circuit tester.

[0033] It is a chemical like the paper-making conditions in the [yield effectiveness trial] applications 1-7 and the examples 1-5 of a comparison. It dilutes with the water which prepared the corrugated paper recycled pulp slurry of 3% of added concentration, and prepared it to the same pH as a pulp slurry to 0.6%. The 500ml TAPPI JOURNAL 56 (10) dynamicdrainage given in 46-50 (OCT.1973) PAPER of the same equipment as Jar RESEARCH MATERIALS and Britt made from INC Jar After flowing into Tester and stirring by 700rpm during 20 seconds, it drains for 10 seconds, and 100ml of filtrate is extracted continuously. The filtrate was filtered through

the filter paper of No.5, and the value of a first pass retention was calculated from the amount of the residue.

[0034]

[Table 2]

		共 重 合 体	比圧縮強度	比破裂強度	裂 断 長 km	C. S. F	OPR %
応 用 例	1	A	18.0	2.63	4.56	390	80.7
	2	B	18.5	2.68	4.62	410	81.8
	3	C	18.7	2.71	4.77	400	81.7
	4	D	18.1	2.65	4.58	400	81.3
	5	E	18.0	2.62	4.56	390	80.8
	6	F	18.2	2.75	4.70	400	82.2
	7	G	18.3	2.75	4.65	395	81.2
応 用 比 較 例	1	H	17.3	2.44	4.25	380	79.0
	2	I	17.6	2.51	4.36	385	80.1
	3	J	17.4	2.57	4.22	380	79.4
	4	K	17.5	2.58	4.38	385	79.9
	5	M	16.6	2.24	4.01	380	79.3

[Translation done.]